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Silica Measurement with High Flow Rate Respirable Size Selective Samplers: A Field Study

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Abstract

High and low flow rate respirable size selective samplers including the CIP10-R (10 l min^{-1}), FSP10 (11.2 l min^{-1}), GK2.69 (4.4 l min^{-1}), 10-mm nylon (1.7 l min^{-1}), and Higgins-Dewell type (2.2 l min^{-1}) were compared via side-by-side sampling in workplaces for respirable crystalline silica measurement. Sampling was conducted at eight different occupational sites in the USA and five different stonemasonry sites in Ireland. A total of 536 (268 pairs) personal samples and 55 area samples were collected. Gravimetric analysis was used to determine respirable dust mass and X-ray diffraction analysis was used to determine quartz mass. Ratios of respirable dust mass concentration, quartz mass concentration, respirable dust mass, and quartz mass from high and low flow rate samplers were compared. In general, samplers did not show significant differences greater than 30% in respirable dust mass concentration and quartz mass concentration when outliers (ratio <0.3 or >3.0) were removed from the analysis. The frequency of samples above the limit of detection and limit of quantification of quartz was significantly higher for the CIP10-R and FSP10 samplers compared to low flow rate samplers, while the GK2.69 cyclone did not show significant difference from low flow rate samplers. High flow rate samplers collected significantly more respirable dust and quartz than low flow rate samplers as expected indicating that utilizing high flow rate samplers might improve precision in quartz measurement. Although the samplers did not show significant differences in respirable dust and quartz concentrations, other practical attributes might make them more or less suitable for personal sampling.

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DISCLAIMER

The findings and conclusions in this report are those of the author(s) and do not necessarily represent the official position of the Centers for Disease Control and Prevention/the Agency for Toxic Substances and Disease Registry.

Keywords

CIP10-R; FSP10; GK2.69; high flow rate samplers; quartz; silica

INTRODUCTION

The US Occupational Safety and Health Administration (OSHA) proposes lowering the permissible exposure limit to 0.05 mg m^{-3} with an action level of 0.025 mg m^{-3} for respirable crystalline silica (RCS) as part of a new comprehensive standard (OSHA, 2014). OSHA's Preliminary Economic Analysis and Initial Regulatory Flexibility Analysis expect a net benefit between 2.8 and 4.7 billion dollars annually over the next 60 years by preventing between 579 and 796 fatalities annually (OSHA, 2013). The mass of RCS on a sample at a specified exposure limit value depends on the sampling time and the flow rate specific to the operation of a particular sampler. The limit of quantification (LOQ) of current analytical methods put constraints on the minimum sample that can be analyzed accurately. Typically, an instrumental LOQ of 10 μg per sample is achievable by the use of either infrared (IR) or X-ray diffraction (XRD) in laboratories maintaining a high standard of quality assurance. In practice, commercial laboratories typically report values >10 μg (OSHA, 2013). The current sampler most commonly used in the USA for OSHA compliance, the 10-mm nylon cyclone, is used with a sampling pump operating at 1.7 l min^{-1}. The 10-mm nylon cyclone can be used to collect samples containing at least 10 μg of respirable quartz at 0.025 mg m^{-3} for 8-h and even 4-h samples, which will allow quantification. However, it cannot be used to quantify concentrations <0.025 mg m^{-3} over periods <8-h sampling periods. In addition, if the proposed OSHA action level is ratified, users would prefer a method able to assure the user that a sample is below this concentration. Performance of respirable size selective samplers operating at high flow rates (flow rate > 4 l min^{-1}) for RCS measurement has been compared to that of low flow rate samplers (1.7–2.2 l min^{-1}) under laboratory and field conditions. These studies have shown that samples collected with high flow rate samplers could provide precise analytical results, i.e. significantly above the limit of detection (LOD) and/or LOQ by increasing the mass collection on filters (Stacey and Thorpe, 2009; Lee *et al*., 2010; Lee *et al*., 2012; Coggins *et al*., 2013; Stacey *et al*., 2014). This study is part of an on-going collaboration between the National University of Ireland, Galway in Ireland and the National Institute for Occupational Safety and Health (NIOSH) in the USA. The objectives of the study are to validate laboratory findings for high flow rate samplers in workplaces.

METHOD

Sampling in the USA

Eight different sites in the USA were included in this study. Work processes underway on these sites included construction (masonry, demolition, and concrete drilling), silica sand production, and metal mining (area sampling only).

Respirable size selective samplers—The two types of low flow rate samplers employed were (i) 10-mm nylon cyclone (Sensidyne, Clearwater, FL, USA), designed for particle collection with 5 μm pore size 37-mm polyvinyl chloride (PVC) filter (GLA5000, SKC Inc., Eighty Four, PA, USA) and a sampling flow rate of 1.7 l min^{-1}, and (ii) Higgins-Dewell type cyclone [model: BGI4L (nickel plated aluminum body and aluminum grit pot), BGI USA Inc., Waltham, MA, USA] used with 5 μm pore size 37-mm PVC filter (GLA5000, SKC Inc.) and a sampling flow rate of 2.2 l min^{-1}. Three high flow rate samplers employed were (i) CIP10-R sampler (Arelco ARC, Paris, France) with particle collection on a polyurethane foam in rotating cup and a sampling flow rate of 10 l min^{-1}, (ii) FSP10 cyclone (GSA Messgerätebau GmbH, Ratingen, Germany) with particle collection on 5 μm pore size 37-mm PVC filter (GLA5000, SKC Inc.) at a sampling flow rate of 11.2 l min^{-1}, and (iii) GK2.69 cyclone (BGI Inc., Waltham, MA, USA), particle collection by 5 μm pore size 37-mm PVC filter (GLA5000, SKC Inc.), sampling flow rate of 4.4 l min^{-1}. In order to minimize particle deposition on sampling cassette walls, conductive polypropylene cassettes (SKC Inc.) were used for 10-mm nylon, BGI4L, and GK2.69 cyclones (Soo *et al*., 2014).

Air sampling and gravimetric analysis—Prior to sample collection, filters and foams for the samplers were equilibrated for a minimum of 72 h in the weighing room at constant relative humidity (50% ± 2) and temperature (26°C ±2). Pre-weighing of filters and rotating cups with foams was performed with a microbalance (XP6U, Mettler-Toledo, Columbus, OH, USA; readability 0.1 μg). Filters and rotating cups with foams were passed through an electrostatic bar (Mettler-Toledo) before weighing to dissipate static charge. A single measurement for each filter and rotating cup was made after allowing exactly 180 seconds for balance stabilization. Average coefficients of variation of blank PVC filters and rotating cups were 0.046 and 0.002%, respectively. The pre-weighed filters were assembled in conductive (static-dissipative) polypropylene cassettes and leak-checked using a field cassette leak tester (SKC Inc.). Aircheck PCXR-4 pumps (SKC Inc.) were used with 10-mm nylon and BGI4L cyclones. SG 10–2 pumps (GSA Messgerätebau GmbH) and Legacy pumps (SKC Inc.) were connected to the FSP10 and GK2.69 cyclones, respectively. The flow rates through the samplers were calibrated using a BIOS DryCal Meter (BIOS International Corporation, Butler, NJ, USA). The flow rates were calibrated before and after each sampling session to confirm that they did not change significantly (all remained within ±5%). The flow rate of the CIP10-R sampler was initially calibrated with a CIP10 Calibration bench (Arelco, ARC) against a tachometer, and the rotational speed of the cup was checked in the field before and after sampling.

Side-by-side personal sampling with six sampler combinations (CIP10-R/10-mm nylon, FSP10/10-mm nylon, GK2.69/10-mm nylon, CIP10-R/BGI4L, FSP10/BGI4L, GK2.69/BGI4L) was conducted with volunteer workers. The participants were asked to wear commercial back-braces (Safe-T-Lift, Style No. 70-110543, FLA Orthopedics Inc. Charlotte, NC, USA) or safety vests (Model SV7O5X, Radians, Memphis, TN, USA) and the high and low flow rate samplers were located in the breathing zone of the worker, one on each side, with sides randomized for different pairs. The pumps were attached to the back-braces around the waist of the participants or in the pockets of vests. Sampling duration was between 10 and 390 min and most samples were collected between 180 and 240 min.

Area sampling was also conducted at four of the sites including metal mining, concrete drilling and construction, and bricklayer training due to the limited number of workers available for personal sampling. A stationary Lippman-type sampling apparatus (Kogut *et al*., 1997; Page *et al*., 2008; Fig. 1) that minimizes spatial variability for area sampling was constructed to allow side-by-side comparison of the high and low flow rate samplers. Five different samplers were placed inside the apparatus while tubing for connection to each sampler to pump was placed outside the apparatus. The on/off switch and air outlet of the CIP10-R sampler were also placed outside the apparatus. The area sampling was utilized when personal sampling was unavailable and the apparatus was placed near working areas at a height of 1.0 m. Area sampling duration was between 78 and 409 min.

The filters and rotating cups with foams were equilibrated in the weighing room for a minimum of 72 h before post-weighing. The respirable dust mass concentration was determined using obtained mass, pre-and post-flow rate, and sampling time.

Sampling in Ireland

Sampling procedures used in Ireland have been described in a previous publication (Coggins *et al*., 2013) and were generally similar to those used in the USA with the following differences: (i) Safety in Mines Personal Dust Sampler (SIMPEDS, Casella, Bedford, UK; particle collection by 5 μm pore size 25-mm PVC filter (GLA5000, SKC Inc.), sampling flow rate at 2.2 l min^{-1}) was used instead of BGI4L cyclone and (ii) personal side-by-side samples were only collected for FSP10/10-mm nylon and FSP10/SIMPEDS pairs due to limited numbers of workers [other pairs were collected as area samples by placing the samplers as near to the worker as physically possible (0.5–15 m from the worker) at a height of 1.5 m using a tripod]. Both SIMPEDS and BGI4L cyclones are based on Higgins-Dewell design and thus similar performance can be assumed (Maynard and Kenny, 1995). Sampling duration was between 15 and 60 min.

Major activities and the number of samples collected for each site are shown in Table 1.

X-ray diffraction analysis

XRD analysis was carried out by an American Industrial Hygiene Association (AIHA) accredited laboratory according to the NIOSH Manual of Analytical Method (NMAM) 7500 [SILICA, CRYSTALLINE, by XRD (filter redeposition)] (NIOSH, 2003), with the exception that samples from CIP10-R sampler were first extracted by adding isopropyl alcohol to the polyurethane foam in its rotating cup, sonicated for 5 min and filtered through a 37-mm PVC filter. Each filter, whether from cyclone or from redeposition of CIP10-R sample, was transferred to a 15 ml vial and 5 ml of tetrahydrofuran (THF) was added. The samples were allowed to stand for 5 min before being placed on a vortex mixer for 2 min. After mixing, the samples were placed in an ultrasonic bath and sonicated for 10 min and then transferred to a silver membrane filter. A silver membrane filter was placed in a vacuum filtration unit. Then, 2 ml of THF was placed on the filter followed by the sample suspension, three vial rinsings, and a final vial cap rinse. Finally, vacuum was applied to deposit the suspension onto the silver membrane filter. The silver membrane filter was then transferred to an aluminum sample plate and placed in the automated sample changer for analysis by XRD (Rigaku Ultima III X-ray diffractometer with D/MAX 2000 PC software). Samples with high levels of respirable dust were analyzed using a dilution procedure. The LOD and LOQ of the laboratory ranged between 5–7 μg and 17–23 μg, respectively. Results between LOD and LOQ were used in the comparisons.

Data analysis

Results of area and personal sampling were combined for each pair of the samplers and data were analyzed using SAS/STAT software, Version 9.3 of the SAS System for Windows (SAS Institute, Cary, NC, USA). Data were transformed using the natural log prior to analysis. Geometric means (GMs) and confidence intervals are back transformed into their natural units for presentation. Sampler types were compared to one another using mixed model analyses of variance carried out with Proc Mixed. Sampling site and sampling pair were considered random variables. Slopes were determined using Proc Reg and making measures from the high flow rate samplers the response variable.

Differences in frequency of below and above LOD and LOQ of quartz mass collected with the high and low flow rate samplers were determined using McNemar's test (McNemar, 1947). All differences were considered significant at $P < 0.05$.

RESULTS

Eleven sets (total of 55 individual samples) of area samples and 268 pairs of personal samples (536 samples) were collected. The ratios of respirable dust concentration, quartz mass concentration, respirable dust mass, and quartz mass between high and low flow rate samplers showed a log normal distribution (Shapiro–Wilk test). These data were described using the GM with 95% levels of confidence. Negative respirable mass, due to low respirable dust mass concentrations, was found in 17 and 6% of samples from low and high flow rate samplers, respectively. Additionally, four samples were lost due to pump failure.

A statistical comparison (McNemar's test) for frequency of below and above LOD (5 μg; NMAM 7500) and LOQ (15 μg; NMAM 7500; a rough estimation from LOD × 3) of quartz mass collected with the high and low flow rate samplers was made (Table 2). The frequency of samples above the LOD was significantly ($P < 0.05$) higher for the CIP10-R and FSP10 samplers compared to low flow rate samplers, while the GK2.69 cyclone did not show significant difference from low flow rate samplers. The same trend was observed in the frequency of results above LOQ between high and low flow rates samplers although frequency above LOQ from the GK2.69 cyclone showed borderline significance compared to low flow rate samplers [$P = 0.059$ and $P = 0.052$ for 10-mm nylon and Higgins-Dewell (HD) type cyclones, respectively].

GM with 95% lower and upper confidence intervals of (i) respirable dust mass concentration ratio, (ii) quartz mass concentration ratio, (iii) respirable dust mass ratio, and (iv) quartz mass ratio for each pair of samplers are shown in Fig. 2. Respirable dust mass concentration and quartz mass concentrations ratios between samplers <0.3 and >3.0 are likely to be outliers caused by field variation rather than bias between sampler performance. The international standards working group for silica measurement (ISO/RC146/SC2/WG7

Silica) compared 13 different respirable size selective samplers including high flow rate samplers that were investigated in the present study in a laboratory environment (calm air condition) and the difference between the samplers were within 60% (Stacey *et al*., 2013). However, it has long been known that work practices, work processes, environmental conditions, and the presence and degree of air movement or ventilation can produce extreme biases between multiple samples from ostensibly the same environment even when identical samplers are used. For example, Van der Wal and Moerkerken (1984) found 'considerable discrepancies in field trials on painters, in which the monitors were worn on opposite lapels under fluctuating conditions of vapor concentration and air movement' and the situation is even more problematic for aerosols where short-distance concentration gradients are commonly encountered through gravitational settling as noted by Vaughan *et al*. (1990) 'The ratio between dust concentrations measured simultaneously on opposite lapels was >2 on >5% of occasions, and is believed to be largely due to real concentration gradients in the environments sampled'. The presence of such outliers can greatly affect the ability to reach a conclusion regarding the similarity of comparisons in an otherwise normal distribution of results. Thus in a study of analytical methods applied to field samples (Bartley *et al*., 2007), a data point was considered an outlier if it was more than three standard deviations from the mean (i.e. where the probability of occurrence in a normally distributed data set is <0.001). In a more recent study of inhalable samplers in the workplace (Lee *et al*., 2011) this criterion was replaced by a simpler exclusion of mass concentration ratios between pairs of samplers <0.3 or >3.0, and the same criterion is applied here. Most outliers in the present study were from Irish samples (site 7, Table 1; >70%) and this is likely because of area sampling near the generation of dust with relatively short sampling times (<60 min) at high respirable dust and quartz concentration. No specific pairing of samplers produced very large number of outliers. Although three pairs of samplers CIP10-R/10-mm nylon, FSP10/10-mm nylon, GK2.69/HD type had the most outliers, there is no consistent pattern indicating a sampler-dependent source.

Since the job tasks in Ireland were similar to each other, they have been grouped as one site. Differences between the sites were not tested for significance because it is a random effect variable rather than fixed effect variable and sample sizes for most sites are too small to have power for statistical analysis.

Sampler pairs of the CIP10-R/10-mm nylon, FSP10/10-mm nylon, and GK2.69/HD type showed significant differences in respirable dust mass concentration and sampler pairs of the CIP10-R/10-mm nylon and GK2.69/HD type showed significant differences in quartz mass concentration (Fig. 2). However, these differences disappeared when outliers were removed from the analysis except the pairs with CIP10-R sampler in quartz mass concentration (Fig. 3). In order to check field variation due to short sampling time in Irish samples, statistical analysis results were compared between samples from all US sites (Irish samples were removed) and samples from all sites when outliers were removed and the results from both groups are similar (Table 3). Ratio of quartz content (%, quartz mass/respirable dust mass × 100) between high and low flow rate flow rate samplers was calculated and its box plot of each pair of samplers is shown in Fig. 4. The pair of CIP10-R and 10-mm nylon samplers only showed significantly different in quartz content (Mann–Whitney rank sum test).

The FSP10 and CIP10-R samplers collected significantly more respirable dust and quartz mass than low flow rate samplers. The GK2.69 cyclone collected significantly more respirable and quartz mass than low flow rate samplers when the outliers were removed from the analysis. The CIP10-R, FSP10, and GK2.69 samplers are expected to collect 5.9, 6.6, and 2.6 times more mass compared to 10-mm nylon cyclone, respectively, and they are expected to collect 4.7, 5.1, and 2.0 times more than HD type cyclone, respectively. GM of respirable dust mass ratios and quartz mass ratios for each pair of the samplers were closer to the expected respirable dust mass ratio and quartz mass ratios without outliers (Fig. 3).

Linear regression analysis results of respirable mass concentrations and quartz concentrations for each pair of samplers with and without outliers are shown in Table 4. Linear regression analysis produced a similar pattern of results to that from mixed model analysis in respirable dust mass concentration and quartz mass concentrations between high and low flow rate samplers. Some sampler pairs showed a significant difference from a 1:1 relationship in respirable dust mass and quartz concentration when outliers were removed (Table 4). However, while the differences were significant, they are not large.

DISCUSSION

Compared to the laboratory study

Performance of high flow rate samplers was previously investigated in laboratory experiments (Lee *et al*., 2010, 2012; Stacey *et al*., 2014) and the high flow rate samplers (CIP10-R, FSP10, and GK2.69) were shown to meet the performance requirement from the International Organization for Standardization (ISO) with respect to respirable convention sampling (ISO, 1995). The samplers showed <30% difference in mass concentration compared to ideal respirable fraction in accordance with a standard protocol (EN13205, 2002). Averages of respirable dust mass concentration and quartz mass concentration ratios comparing high and low flow rate samplers from laboratory studies (Lee *et al*., 2012; Stacey *et al*., 2014) and from the present study are shown in Table 5. Although the average ratios from the laboratory studies are based on arithmetic means with standard deviations and those from the present study are GMs and upper and lower 95% confidence intervals without outliers, the average ratios from the studies are comparable in both respirable dust mass concentration and quartz mass concentration. Comparison by linear regression analysis of respirable dust mass concentration between laboratory and field studies is shown in Table 6, where the intercept is forced through zero (slopes in Tables 4 and 6 from the present study are different due to this difference in intercept). Difference between the slopes of respirable dust mass concentration and quartz mass concentration are generally smaller than difference in average of ratios between samplers. While the average quartz mass concentration from the CIP10-R sampler is significantly larger than that from 10-mm nylon and HD type cyclones (Fig. 4), linear regression analysis showed that the CIP10-R sampler provided significantly lower quartz mass concentration than HD type cyclone (Table 4), which has been observed previously (Stacey and Thorpe, 2009; Stacey *et al*., 2013; Verpaele and Jouret 2013; Stacey *et al*., 2014).

Practical considerations

While the most important consideration in air sampling is the accuracy and precision in measuring the intended size fraction, i.e. inhalable, thoracic, or respirable, there are other important issues including cost of sampling, worker acceptance (comfort and placement of sampling device) and industrial hygienist concerns (ease of calibration and sample analysis; for example, the jar necessary for calibration of the 10-mm nylon cyclone is cumbersome in the field). The FSP10 cyclone has been calibrated to provide a respirable sample at 11.2 l min^{-1}, which would provide adequate sensitivity for most purposes (Lee *et al*., 2010; Stacey *et al*., 2014), even for shorter task-based sampling at these concentrations. However, the cyclone might be considered bulky and heavy and so are the personal pumps necessary for its operation. Workers, who are not used to such encumbrance, have resisted wearing them (Stacey and Thorpe, 2009; Coggins *et al*., 2013) but it may be subjective observation based on prior habituation with low flow pumps and smaller cyclones. A GK4.162 cyclone (BGI Inc.), a natural extension of the GK2.69, was recently developed and it operates at a higher flow rate, 8.5 or 9.5 l min^{-1} (Thorpe, 2011). However, the size and weight of the cyclone are similar to those of the FSP10 cyclone and larger and heavier pumps are still necessary for its operation. The CIP10-R sampler is relatively light weight compared to other cyclones. Samples from the CIP10-R sampler requires an extra sample preparation procedure for quartz analysis and the sampler underestimated concentrations compared to other samplers in previous laboratory studies—up to 35% for respirable dust concentration plus another 10–15% for silica measurement, along with larger variability in the weighing procedure (Stacey and Thorpe, 2009; Stacey *et al*., 2014). The differences between the CIP10-R sampler and other samplers is atributed to the unique sampling efficiency curve of the sampler (Courbon *et al*., 1988; Görner *et al*., 2001; Lee *et al*., 2010) and it would be dependent on the size distribution of the workplace aerosols, which may be the reason for unusually higher mass concentrations in the present study. The cost of the high flow sampling equipment is relatively high compared to low flow rate sampling equipment (Stacey and Thorpe, 2009). Moderate flow rate cyclones such as the GK2.69 cyclone represent a reasonable middle ground between the size and weight associated with high flow rate samplers and potentially inadequate collection of respirable quartz mass with low flow rate samplers. This cyclone will provide a respirable sample at 4.2 or 4.4 l min^{-1}, and therefore provide a sample loading of 12 or 13 µg RCS at a concentration one-half of 0.025 mg m^{-3} over a 4-h sample. Since it uses a 37-mm filter, the pressure-drop is within the range of some lower-cost personal sampling pumps already in common use for taking an 8-h sample. The GK2.69 cyclone showed no significant difference in number of samples above the LOD and borderline significance above the LOQ compared to low flow rate samplers (Table 2) in the present field study while samples from the GK2.69 showed a higher frequency above LOD and LOQ compared to 10-mm nylon cyclone in the laboratory study (Lee *et al*., 2012). The GM of respirable dust and quartz mass were significantly lower in pair with HD type cyclones from personal sampling (Fig. 2), this difference disappeared with removal of outliers and performance matched previous laboratory studies (no significant differences in respirable dust and quartz mass concentration and around two times of net mass collection; see Fig. 3). The outliers in results using the GK2.69 and HD type cyclones might be attributable to the large field variation and short sampling times (<60 min) in high respirable dust or quartz concentration found on the Irish situations rather than due to the performance of GK2.69 cyclone as described above. Higher flow rates also increase the filter mass collected, which could lead to samples losses. However, a round-robin filter weighing exercise carried out in the UK did not show losses of up to 4-mg sample loading on PVC and glass-fiber filters transported by regular mail (McLister *et al*., 2001; Stacey *et al*., 2013). While 2 mg has been cited as an upper limit to the maximum filter loadings to minimize X-ray absorption effects, 4 mg can be accommodated with appropriate calibration (Mecchia *et al*., 2013). Thus a filter loading of 4 mg is probably a more reasonable upper limit to the collection of respirable dust for silica analysis. The sample matrix affects the analysis and limits the determination of silica in samples containing <1%. A target concentration of 0.025 mg m^{-3} has a consequence of imposing a limit for the respirable fraction of particles not otherwise specified (or regulated) of 2.5 mg m^{-3}. However, a full-shift sample of respirable dust with a GK2.69 cyclone at 2.5 mg m^{-3} will approximately have a mass of 5 mg, so it may be prudent to restrict high flow rate sampling in very dusty environments.

Variation in RCS analysis determined from the AIHA Proficiency Analytical Testing (PAT) results between 2005 and 2013 [average relative standard deviation (RSD) 20%] was reduced compared to the period between 1990 and 1998 (average RSD 29%), but it is still higher than for other occupational samples (Harper *et al*., 2014). Previous studies of PAT results showed a pronounced trend toward even higher standard deviations at the lower loadings that would result from using cyclones with flow rates around 2 l min^{-1} to collect samples at the proposed action limit. The new analysis suggested this trend was a result of sample preparation procedure, rather than analytical capability, but it remains the case that low flow rates at the action level concentration produce sample loadings below the current range of PAT samples. High flow cyclones provide higher loadings, which would fall within the PAT sample range.

CONCLUSIONS

Performance of high flow rate samplers for respirable size selective sampling including CIP10-R, FSP10, and GK2.69 was compared to that of low flow rate samplers in occupational environments. The high flow rate samplers did not generally show significant differences in respirable dust and quartz mass concentration. The high flow rate samplers allow for greater respirable quartz mass collection over shorter sampling periods affording improved levels of precision. However, higher flow rate samplers may have other attributes including cost and size and weight of both sampler and pump that may influence the decision as to whether to use them for routine personal sampling. A cyclone operated at 1.7 l min^{-1} for 4 h at the proposed action level would collect 10 µg of silica, which is the limit of quantitation for many laboratories. The present study confirms the conclusions of previous studies that respirable size selective samplers operating with high flow rate can be used to reliably quantify silica concentrations below 0.025 mg m^{-3} over sampling periods <8 h or 0.025 mg m^{-3} for sampling periods <4 h.

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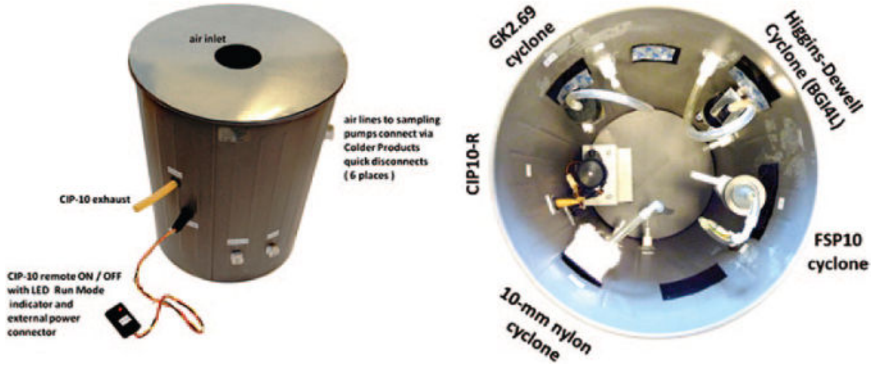

Figure 1.
Area sampling apparatus for collection of respirable dust.

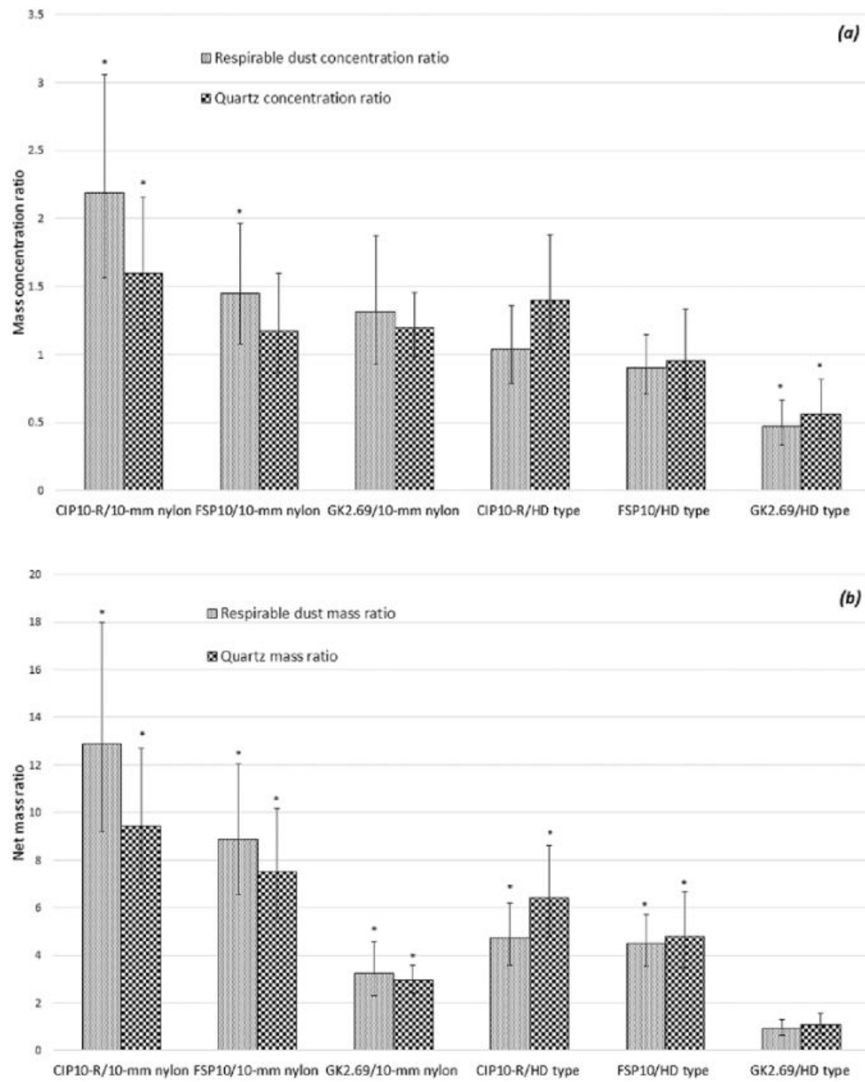

Figure 2.
Geometric means (95% lower and upper confidence intervals) of high/low flow rate (a) mass concentration and (b) net mass ratios including outliers. *Significantly difference between two samplers in accordance with mixed model analyses of variance ($P < 0.05$). Sample number is between 33 and 49. HD type is Higgins-Dewell type.

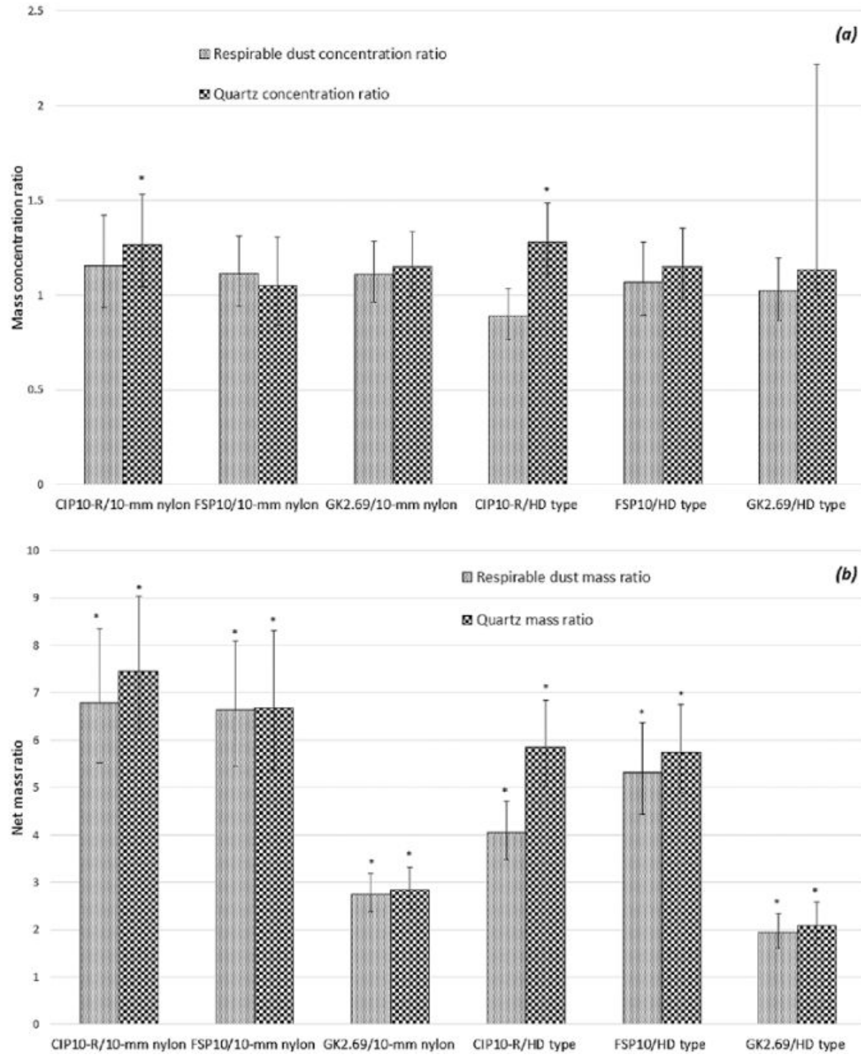

Figure 3.

Geometric means (95% lower and upper confidence intervals) of high/low flow rate (a) mass concentration and (b) net mass ratios without outliers. *Significantly difference between two samplers in accordance with mixed model analyses of variance ($P < 0.05$). Sample number is between 24 and 41. HD type is Higgins-Dewell type.

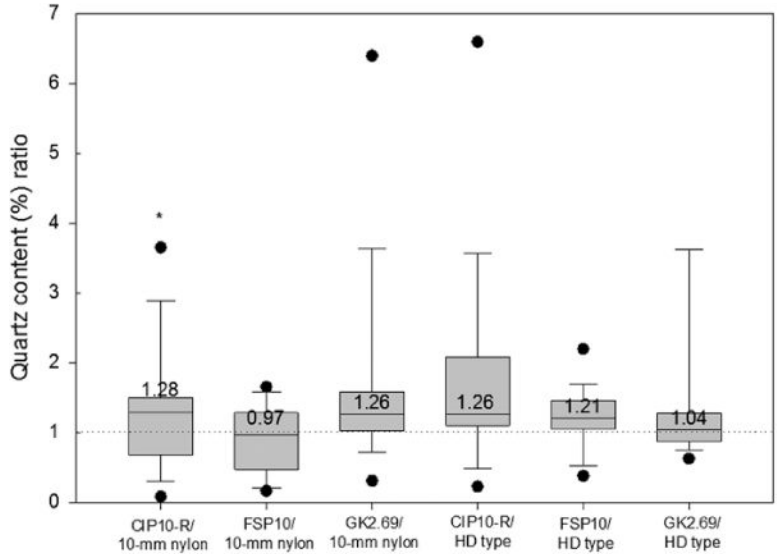

Figure 4.

Box plot of quartz content ratio between high and low flow rate samplers. The horizontal lines in the box plot from bottom to top indicate 10th, 25th, 50th (median), 75th, and 90th percentiles. The circles indicate the 5th (lower circle) and 95th (upper circle) percentiles. HD type is Higgins-Dewell type. *Significantly difference between two samplers in accordance with Mann–Whitney rank sum test ($P < 0.05$).

Table 1

Sampling site, major process, number of sample pairs collected, number of outliers for each sites samples, respirable dust mass [geometric mean (95% lower and upper confidence interval)], and quartz mass concentration [geometric mean (95% lower and upper confidence interval)] for each site

Sampling site	Major process	Number of sample collected (pairs)	Number of outliers in ratios concentration (pairs)		Respirable dust mass concentration (mg m^{-3})	Quartz mass concentration (μg m^{-3})
			Quartz	Respirable dust		
1 Masonry US	Cutting, tuck-pointing	22	5	5	0.350 (0.224–0.548)	63.5 (43.1–93.7)
2 Demolition US	Jack hammering	28	5	6	0.096 (0.064–0.144)	14.8 (8.53–25.5)
3 Bricklayer training center US	Cutting, tuck-pointing	8	2	2	1.07 (0.322–3.57)	216 (57.7–805)
4 Construction training center US	Drilling	12	0	0	2.24 (0.906–5.52)	304 (128–721)
5 Sand production US	Crushing, mining, milling, loading	54	2	7	0.097 (0.074–0.129)	18.9 (15.4–23.1)
6 Sand production US	Bagging, milling, loading	30	1	5	0.085 (0.068–0.106)	45.6 (34.5–60.4)
7 Restoration stonemasonry Ireland	Cutting	114	45	52	7.45 (5.84–9.51)	4062 (2722–5881)

Table 2

Frequency of quartz mass below and above limit of detection and limit of quantification collected with high and low flow rate samplers

Pair of samplers	Sample number below LOD[2]	Sample number above LOD[2]	*P* value[3]	Sample number below LOQ[4]	Sample number above LOQ[4]	*P* value[3]
10-mm nylon	19	36	<0.0001	30	25	<0.0001
CIP10-R	3	52		9	46	
10-mm nylon	21	35	0.0008	32	24	<0.0001
FSP10	8	48		13	43	
10-mm nylon	18	37	0.103	26	29	0.059
GK2.69	14	41		21	34	
HD type[1]	11	43	0.033	21	33	0.0005
CIP10-R	5	49		9	45	
HD type[1]	16	40	0.0039	25	31	0.0002
FSP10	6	50		11	45	
HD type[1]	16	41	0.317	28	29	0.052
GK2.69	14	43		21	36	

[1] Higgins-Dewell type, including BGI4L and SIMPEDS.

[2] Limit of detection.

[3] *P* value from McNemar's test.

[4] Limit of quantification.

Table 3

Comparison of linear regression analysis of respirable mass concentration and quartz concentration between samples from all US sites (Irish samples were removed) and samples from all sites when outliers were removed

Pair of samplers	Respirable dust mass concentration		Quartz concentration	
	Samples from all US site (Irish sample were removed)	**Sample for all site (outliers removed)**	**Sample from all US site (Irish sample were removed)**	**Sample from all site–8 (outliers removed)**
CIP10-R/10-mm nylon	1.04	1.08	1.02	0.96
FSP10/10-mm nylon	0.94	1.02	0.90	1.05
GK2.69/10-mm nylon	0.97	1.03	0.91*	1.08*
CIP10-R/Higgins-Dewell type	1.02	0.94	1.01	0.89*
FSP10/Higgins-Dewell type	1.01	0.92*	1.01	0.96
GK2.69/Higgins-Dewell type	0.94	0.99	0.94	1.01

*Significantly different from 1:1 relationship ($P < 0.05$).

Table 4

Linear regression analysis of respirable mass concentration and quartz concentration for each pair of samplers with and without outliers

Samplers pair	Slope (R^2) w/respirable dust mass concentration	Slope (R^2) w/respirable dust mass concentration (outlier removed)	Slope (R^2) w/quartz mass concentration	Slope (R^2) w/quartz mass concentration (outlier removed)
CIP10-R/10-mm nylon	0.89 (0.71)	1.08 (0.94)	0.89 (0.81)	0.96 (0.93)
FSP10/10-mm nylon	1.1 (0.84)	1.02 (0.95)	1.2 (0.92)*	1.05 (0.94)
GK2.69/10-mm nylon	0.89 (0.72)	1.03 (0.96)	1.1 (0.96)*	1.08 (0.97)*
CIP10-R/Higgins-Dewell type	0.85 (0.84)*	0.94 (0.96)	0.77 (0.89)*	0.89 (0.98)*
FSP10/Higgins-Dewell type	0.87 (0.89)*	0.92 (0.95)*	0.87 (0.87)*	0.96 (0.97)
GK2.69/Higgins-Dewell type	0.68 (0.81)*	0.99 (0.95)	0.66 (0.85)*	1.01 (0.96)

*Significantly different from 1:1 relationship ($P < 0.05$)

Table 5

Comparison of average respirable dust mass concentration ratio and quartz mass concentration ratios between high and low flow rate samplers. The average ratios from the laboratory studies (Lee *et al*., 2012; Stacey *et al*., 2014) are based on arithmetic means with standard deviations and those from the present study are geometric means and upper and lower 95% confidence intervals without outliers.

Pair of samplers	Lee *et al*. (2012)		Stacey *et al*. (2014)	Present study	
	Respirable dust mass concentration ratio	Quartz mass concentration ratio	Quartz mass concentration ratio	Respirable dust mass concentration ratio	Quartz mass concentration ratio
CIP10-R/10-mm nylon	1.10 (0.193)	1.17 (0.555)		1.15 (0.936–1.42)	1.26 (1.04–1.53)
FSP10/10-mm nylon	1.22 (0.168)	1.46 (0.762)		1.11 (0.945–1.31)	1.05 (0.841–1.31)
GK2.69/10-mm nylon	1.09 (0.183)	1.32 (0.602)		1.11 (0.960–1.28)	1.15 (0.995–1.34)
CIP10-R/Higgins-Dewell type	1.03 (0.207)	1.05 (0.504)	0.88	0.891 (0.766–1.04)	1.27 (1.09–1.48)
FSP10/Higgins-Dewell type	1.14 (0.172)	1.27 (0.535)	1.07	1.07 (0.891–1.28)	1.15 (0.977–1.35)
GK2.69/Higgins-Dewell type	0.999 (0.171)	1.13 (0.412)	1.00	1.02 (0.867–1.19)	1.13 (0.997–1.29)

Table 6

Linear regression analysis comparison in respirable dust mass concentration between high and low flow rate samplers

Samplers pair	Lee *et al*. (2012)	Stacey *et al*. (2014)	Present study
CIP10-R/10-mm nylon	1.02	1.01	1.12
FSP10/10-mm nylon	1.19	1.21	1.05
GK2.69/10-mm nylon	1.06	1.03	1.04
CIP10-R/Higgins-Dewell type	0.96	0.84	0.97
FSP10/Higgins-Dewell type	1.14	0.99	0.98
GK2.69/Higgins-Dewell type	1.02	0.84	0.95